

**~~DRAFT~~ FINAL QUALITY ASSURANCE PROJECT
PLAN**

for

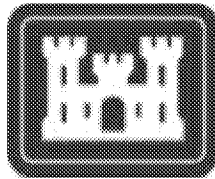
PASSIVE SAMPLING

at

**River Operable Unit, Bradford Island
CASCADE LOCKS, OREGON**

Prepared by

U.S. ARMY CORPS OF ENGINEERS
Portland and Seattle Districts



~~December 2~~ January 23, 2020 ~~19~~

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TITLE AND APPROVAL SHEET
QUALITY ASSURANCE PROJECT PLAN (QAPP)
PASSIVE SAMPLING
RIVER OU, BRADFORD ISLAND, CASCADE LOCKS, OREGON

This Quality Assurance Project Plan (QAPP) describes sampling activities and Data Quality Objectives (DQOs) for passive sampling at the River OU, Bradford Island, Cascade Locks, OR. The QAPP is based on the *Intergovernmental Data Quality Task Force Uniform Federal Policy for Quality Assurance Project Plans Guidance (EPA 2009)*.

Chris Budai, Project Manager, NWP

Date

William Gardiner, Study Technical Lead, NWS

Date

Alison M. Suess, Ph.D., Chemist, NWS

Date

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TABLE OF CONTENTS

1. PROJECT MANAGEMENT AND OBJECTIVES.....	9
1.1. Project Organization, Responsibilities and Authority	9
1.1.1. Communication Pathways.....	10
1.1.2. USACE Personnel Responsibilities and Qualifications	11
1.1.3. Technical Advisory Group Personnel Responsibilities.....	12
1.2. Data Quality Objectives and Measurement Performance Criteria.....	12
1.2.1. Development of Data Quality Objectives Using the Systematic Planning Process	12
1.2.2. Subset of PCB Congeners for Analysis.....	18
1.2.3. Statistical and Geospatial Analysis for Determination of Primary Source Material.....	20
1.2.4. Temperature Monitoring to Assess Groundwater Influence	21
1.2.5. Measurement Performance Criteria	21
1.3. Secondary Data Evaluation.....	21
1.4. Project Overview and Schedule.....	21
2. DATA GENERATION AND ACQUISITION.....	23
2.1. Sampling Tasks.....	23
2.1.1. LDPE Sampling Apparatus.....	23
2.1.2. Temperature Data Loggers.....	23
2.1.3. Sampler Deployment and Retrieval	23
2.1.4. LPDE Sampler Field Processing.....	26
2.1.5. Decontamination Procedures	28
2.1.6. Field Equipment Calibration, Maintenance, Testing and Inspection Procedures	28
2.1.7. Supply Inspection and Acceptance Procedures.....	28
2.1.8. Field Documentation Procedures	28
2.1.9. Sample Delivery.....	29
2.1.10. Sample Custody	29
2.1.11. Disposal of Investigative Derived Wastes	29
2.2. Analytical Tasks	29
2.2.1. Analytical Methods	30
2.2.2. Analytical Instrument Calibration Procedures	30
2.2.3. Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures..	30
2.3. Quality Control Samples	30

2.3.1.	Field Quality Control Samples.....	31
2.3.1.1.	Field Quality Control Samples.....	31
2.3.1.2.	Field Duplicates	31
2.3.1.3.	Trip Blanks	31
2.3.1.4.	Field Blanks	31
2.3.2.	Analytical Method Quality Control Samples.....	31
2.3.2.1.	Method Blanks.....	31
2.3.2.2.	Laboratory Control Samples (LCS)	31
2.3.2.3.	Matrix Spike and Matrix Spike Duplicate (MS/MSD).....	31
2.3.2.4.	Surrogates	32
3.	ASSESSMENT AND OVERSIGHT	32
4.	DATA MANANGEMENT AND DOCUMENTATION.....	33
4.1.	QAPP	33
4.2.	Final Report.....	33
4.3.	Laboratory Documentation (Data Package Deliverables)	33
4.3.1.	Data Package Deliverables.....	33
4.3.2.	Electronic Data Reporting Formats.....	33
5.	DATA REVIEW, VERIFICATION, AND VALIDATION.....	34
5.1.	Review of Laboratory Data	34
5.2.	Data Verification and Validation Stages	34
5.2.1.	Stage 1.....	34
5.2.2.	Stage 2A.....	35
5.2.3.	Stage 2B	36
5.2.4.	Stage 3.....	37
5.2.5.	Stage 4.....	38
5.3.	Data Verification and Validation Stages	38
5.4.	Usability Assessment.....	39
6.	REFERENCES	39

APPENDICIES

Appendix A: Operating Procedure for the Preparation, Extraction, and Analysis of Low Density Polyethylene used as a Passive Sampling Technique in Sediment and Surface Waters

Appendix B. Sample Coordinates

Appendix C. Sample Location Maps

Appendix D. Field Form

Appendix E. Vessel Positioning, Sampler Schematic, Sampler Prototype Photos

Appendix F: Activity Hazard Analysis (AHA)

LIST OF ACRONYMS

µg/L	microgram per liter
AOPC	area of potential concern
CaCO ₃	calcium carbonate
CCB	continuing calibration blank
CCV	continuing calibration verification
CoC	chain of custody
COPC	contaminant of potential concern
CPR	cardiopulmonary resuscitation
DoD ELAP	Department of Defense Environmental Laboratory Accreditation
DoD QSM	Department of Defense Quality Systems Manual
DMC	deuterated monitoring compounds
EDD	electronic data deliverables
EDTA	ethylenediaminetetraacetic acid
EPA	United States Environmental Protection Agency
GC-MS	gas chromatography mass spectroscopy
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	high density polyethylene
HNO ₃	nitric acid
HPAH	high-molecular-weight polycyclic aromatic hydrocarbon
ICB	initial calibration blank
ICV	initial calibration verification
JHA	Job Hazard Analysis
LCS	laboratory control sample
LOD	limit of detection
LOQ	limit of quantitation
mg/kg	milligram per kilogram
MS	matrix spike
MSD	matrix spike duplicate
ODEQ	Oregon Department of Environmental Quality
OU	Operable Unit
PCB	polychlorinated biphenyl
PDT	Project Delivery Team
POC	point of contact
PM	Project Manager
PQO	Project Quality Objectives
QC	quality control
RI	Remedial Investigation
RL	reporting level
SLV	screening level value
SOP	Standard Operating Procedure
TAG	Technical Advisory Group
UCL	upper confidence limit
UPL	upper prediction limit
USACE	United States Army Corps of Engineers
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
WP-QAPP	Work Plan with Quality Assurance Project Plan

1. PROJECT MANAGEMENT AND OBJECTIVES

1.1. Project Organization, Responsibilities and Authority

The Project Delivery Team (PDT) for this Work Plan (WP) includes members from USACE Portland and Seattle Districts.

The project team provides the overall framework for the data collection approach by defining project objectives and data quality requirements, and ensuring that they are met during the execution of the project. Project updates will be shared with the Technical Advisory Group (TAG) who will be coordinated with during development and provided final copies of the WP and QAPP by the USACE Project Manager (USACE PM). This section further describes the team project roles. Figure 1 and Table 1 present the project organization.

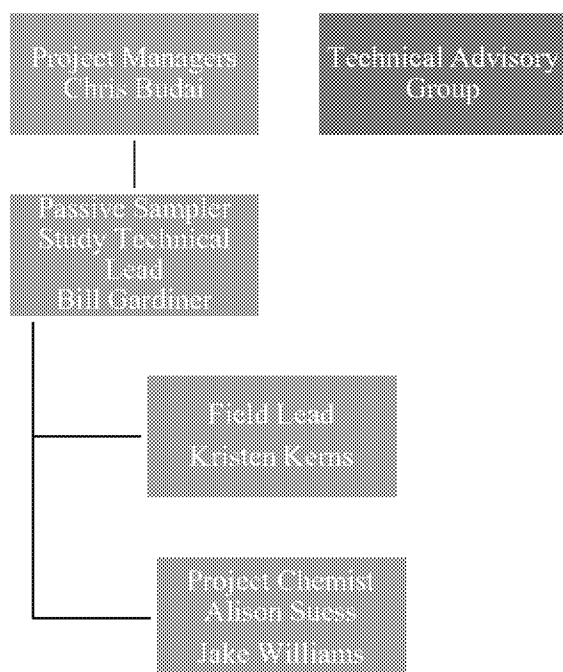


Figure 1. Project Organization Chart

Table 1. Project Organization and Distribution List

Personnel	Contact Information	Title
USACE		
Chris Budai	333 SW 1st Ave Portland, OR 97204 Phone: 503-808-4725 Email: christine.m.budai@usace.army.mil	Project Manager
William Gardiner	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3322 william.gardiner@usace.army.mil	Study Technical Lead
Alison M. Suess, Ph.D.	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3264 alison.m.suess@usace.army.mil	Project Chemist
Kristen Kerns	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3474 kristen.kerns@usace.army.mil	Field Lead

1.1.1. Communication Pathways

Communication is a key to the success of this project. Communication pathways describe the points of contact for resolving sampling and analysis problems, for distributing data to users, soliciting concurrence and obtaining approval between project personnel and contractors. Communication pathways are summarized in Table 2.

Table 2. Communication Pathways

Communication Driver	Responsible Entity	Name Phone Number	Procedure (timing, pathway, etc.)
USACE management for this project Overall direction and Point of Contact for public	Project Manager	Chris Budai 503-808-4725	Assures that the overall direction of the project is consistent with USACE guidance Liaison with the Public
QAPP approval	Technical Lead	Bill Gardiner 206-764-3322	Coordinates with Project Manager, Project Lead, Chemist and Field Lead on project technical issues
Schedule, budget and technical issues			Reports to USACE PM regarding schedule, budget, and technical issues
Changes to schedule and budget			Notifies USACE PM of significant changes in execution or schedule
Oversight of final report Provides coordination among team members			Oversee USACE writing of final report and distribution to reviewers Provides input to QAPP and data reports

Communication Driver	Responsible Entity	Name Phone Number	Procedure (timing, pathway, etc.)
Writes QAPP with input from technical team members. Laboratory and data validation	Project Chemist	Alison M. Suess, Ph.D. 206-764-3264 Jake Williams 206-316-3157	Oversees writing of QAPP and Activity Hazard Analysis (AHA) and ensures revision approval within agreed timeframe Oversees laboratory work Writes data validation report Provides laboratory and data validation components of QAPP
Provide direction to field teams on sample collections Delivery of samples to laboratory Sampling activities summary Ensures compliance with Site Safety Health Plan (SSHP) AHA	Field Lead	Kristen Kerns 206-764-3474	Daily communication with team members during sampling events Coordinates with Project Chemist and laboratory for sample delivery Documents all field activities in Final Monitoring Report Briefs field team on SSHP and JHA and documents noncompliance Coordinates with Project Chemist

1.1.2. USACE Personnel Responsibilities and Qualifications

USACE Project Manager

The project manager (PM), Chris Budai, is responsible for the execution of the scope, schedule, and budget for the Bradford Island CERCLA project. She is the primary POCs for communications with stakeholders. The USACE PM also has the authority stop work of USACE staff. The USACE PM is the primary document controller for the WP.

USACE Technical Lead

The Technical Lead, Bill Gardiner, will oversee all activities of the USACE project delivery team (PDT), including quality assurance reviews, and maintain regular coordination to ensure adequate and timely flow of information for all work.

USACE Project Chemist

The Project Chemists, Alison M. Suess, Ph.D. and Jake Williams, are directly responsible for laboratory coordination and matters related to chemistry. They are responsible for providing additional guidance to the Field Sampling Lead (Kristen Kerns) in any matters relating to project chemistry and data quality.

Field Sampling Lead/Site Health and Safety Officer

Kristen Kerns is the designated field sampling lead and site safety and health officer (SSHO) for this effort. She is responsible for coordinating the sampling with relevant Bonneville Project staff, execution of sampling, and shipping of samples to the project laboratories. She may communicate directly with the PM, Technical Lead, and Project Chemists as needed during the field sampling event.

Special Training Requirements and Certifications

Project staff shall be qualified to perform their assigned jobs. Field sampling personnel conducting or monitoring sampling activities are to be trained by the field sampling lead in accordance with established USACE protocols.

Field Staff

All project staff participating in on-site field activities shall have current HAZWOPER training in accordance with 29 Code of Federal Regulations (CFR) Part 1910.120. The field sampling lead has HAZWOPER training in accordance with the same standard as well as a current certification in first aid and CPR.

Laboratory Contact

The analytical laboratories and applicable information that will be used for this project are listed below in Table 3.

Table 3. Analytical Laboratory and Contacts

Lab Name	Lab Address	POC	Contact Info	Role
Texas Technical Institute	2500 Broadway Lubbock, TX 79409-0000	Dr. Danny Reible	806-834-8050 Danny.Reible@ttu.edu	Technical Lead
		Alex Smith	Alex.V.Smith@ttu.edu	Graduate Student

1.1.3. Technical Advisory Group Personnel Responsibilities

Technical Advisory Group members represent their respective agencies and provide technical review of the QAPP.

1.2. Data Quality Objectives and Measurement Performance Criteria

1.2.1. Development of Data Quality Objectives Using the Systematic Planning Process

As described in the Final Work Plan (USACE, 2019), the goal of this study is to support remedial design in the River OU by identifying areas along the northern shoreline of Bradford Island that may still be serving as a primary source of PCB contamination to fish and other aquatic receptors and to identify those areas that may not be an ongoing source of PCBs. Measuring concentrations of PCBs in water at the sediment-water interface (porewater and near-bottom water) will be used to identify source areas.

The two primary goals of this study are:

- 1) Identify locations along the northern and eastern tip of Bradford Island that are potential source areas.
- 2) Use passive sampling results as a line of evidence to eliminate source areas along the northern and eastern tip of Bradford Island.

To support these overall goals, Data Quality Objectives (DQOs) were developed through the systematic planning process as described in the UFP-QAPP Guidance. This section presents the DQOs for the passive sampler program. The DQO process defines criteria that will be used to establish the final data collection design (U.S. EPA 2006). Based on the study goals listed above, the DQOs were developed to support the selection of sampling and analysis methods and an overall study design that leads to data appropriate to answer the study questions. The DQOs developed for the passive sampler study, the data types, and the analytical approaches are presented in the following subsections and are summarized on Table 4. Specific performance goals, referred to as Data Quality Indicators, for the individual analytical methods are discussed in Section 3.0 after the methods have been introduced.

DQO-1: Identify locations that are ongoing sources of PCBs at Bradford Island. The first DQO is to determine whether there are ongoing source areas present in the River OU north and east of Bradford Island. This first DQO establishes individual locations or localized areas that are acting as a source of PCBs to the sediment and biota of Bradford Island. Source areas are those locations with concentrations of total PCBs that are highly concentrated, highly mobile, or not reliably containable. In general, PCBs associated with the legacy waste that may still be present in the River OU are unlikely to be highly mobile; however, they may be “highly concentrated” and “not reliably containable”.

Investigative methods to determine whether there are locations that have PCBs that are highly concentrated or not reliably contained will include measurement of freely dissolved PCBs in water at the sediment-water interface (the porewater and near-bottom water). PCB congeners will be measured using passive samplers that are placed in situ for a minimum of 28 days. A total of 163 low-density polyethylene (LDPE) passive samplers will be deployed at a high density across the study area. Individual locations or groups of locations that have PCB concentrations that are elevated relative to the surrounding area will be considered source areas in the remedial design.

Source areas will be identified for total PCBs (based on the sum of 45-46 selected congeners) based on ~~three several~~ types of analysis: 1) points identified using Grubb’s outlier test and/or Q-Q plots to help graphically illustrate potential outliers and population partitioning methods to evaluate whether distinctly separate groups of congeners are present; and/or 2) points fitting the ODEQ definition of hot-spots source material (10 times the 90UCL); ~~or~~ and/or 3) groups of stations identified as significantly elevated through geostatistical analysis (kriging).

DQO-2: Identify locations that may not be ongoing sources of PCBs at Bradford Island. The second DQO is to determine areas that are not sources in the River OU north and east of Bradford Island. This second DQO establishes individual locations or localized areas that *may not* be acting as a source of PCBs to the sediment and biota of Bradford Island. This DQO allows remedial design to adjust remedy alternatives that are not targeted to source areas (areas that are highly contaminated or with PCBs that are not reliably contained).

Investigative methods to determine whether there are locations that may not be source areas are similar to those for DQO-1, with the exception that a negative result using passive samplers will be considered a first step, to be followed by sediment and biota sampling.

DQO-3: Identify locations that may represent an area of groundwater upwelling at Bradford Island.

This final DQO is to determine the potential for groundwater upwelling along the northern and eastern portion of Bradford Island. To do this, time series graphs of temperature readings during deployment will be compared to time series graphs of surface water temperature readings collected at the same time. Significant differences in temperature between surface water and individual sediment water interface may be indicative of groundwater upwelling. This may ultimately aid in the interpretation of PCB results obtained through collocated passive sampling.

Table 4. Data Quality Objectives

<i>DQO Step</i>	<i>DQO 1</i>	<i>DQO 2</i>	<i>DQO 3</i>
1. State the problem	PCB concentrations in Bradford Island biota and sediment remain elevated following sediment removal in 2007 suggesting the potential for an ongoing source to the River OU. In order to complete the remedial design for the River OU, locations that may be acting as a source, as well as those areas that are not acting as a source, need to be identified. However, the complex bottom topography and lack of bedded sediment do not support the use of conventional sediment sampling methods to identify sources.		It is unknown whether groundwater upwelling may have an influence on PCB concentrations measured using passive sampling.
2. Identify the goals of the study	Identify locations that ARE ongoing sources of PCBs at Bradford Island	Identify locations that MAY NOT be ongoing sources of PCBs at Bradford Island	Identify groundwater upwelling locations
3. Identify the information inputs	Concentrations of PCB congeners at the sediment-water interface	Concentrations of PCB congeners at the sediment-water interface	Individual point samples of time series water temperature readings at the sediment-water interface and surface water
4. Define the boundaries of the study	Northern and eastern portions of Bradford Island – areas with legacy waste and previously observed elevated PCB levels as described in Section 2.1.3		
5. Develop the analytical approach	Outlier test (Grubb's Test); <u>population partitioning</u>	Outlier test (Grubb's Test); <u>population partitioning</u>	Analysis of difference between time series temperature results for sediment water interface at individual point samples with collocated passive samplers versus surface water.
	Ten times 90UCL (ProUCL)	Ten times 90UCL (ProUCL)	
	Geospatial analysis	Geospatial analysis	
6. Specify performance or acceptance criteria	Performance or acceptance criteria are described in Section 2, including field QC samples and laboratory QC samples. DQIs for laboratory analyses will be met, as described in Section 2		Individual temperature measurements at the sediment water interface and surface water samplers sufficient to develop a time series plot and calculate an average at each individual point sample with a recoverable collocated passive sampler.
7. Develop the detailed plan for obtaining data	LDPE passive samplers will be deployed on the sediment surface for 28 days at 163 sampling locations that have been selected based on a systematic sampling design with a triangular grid and a random start. Each sampler will be extracted and extracts analyzed for 45-46 PCB congeners. A full list of 209 total quantifiable congeners will be measured in extracts from 10 samplers. Equilibrium concentrations will be calculated for each congener using performance reference compounds and results present by congener and as total PCB congeners based on the list of 45 congeners		Individual temperature sensors will be collocated with passive samplers during deployment. Five temperature sensors suspended mid water column will also be deployed. Temperature readings will be recorded at a minimum of every hour during the equilibration period then retrieved with the passive samplers at the end of equilibration. Temperature data will be downloaded from sensors and analyzed for any correlation with PCB concentrations measured in passive samplers.

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Table 5. Sample Locations, Media, Methods, Analytes of Interest, and Detection and Reporting Limits

Sample Locations and Media	Method	Analytes	LOD*	LOQ/RL*
River OU, LDPE	GC-TQMS (Agilent 7890B) using SIM/SIM mode (EPA Method 1668c)	Subset of PCB Congeners	≤1 ng/g PE	≤1 ng/g PE

*Detection limits (LOD) and reporting limits (RL; also known as limit of quantitation (LOQ) are estimated and may change due to specific laboratory conditions, for example, dilutions.

*To achieve the desired reporting limits, the laboratory will run a modified method 8082A for congener analysis. Note: This method is not compliant with DoD-QSM-5.1 and it is not DoD-ELAP accredited.

1.2.2. Subset of PCB Congeners for Analysis

As discussed in the Final Work Plan, each individual PE sampler will be analyzed for a group of PCB congeners. To allow for an increased number of sampling locations, the congener list was refined to include those congeners that are significant contributors to the PCBs observed in Bradford Island media (sediment, tissues, porewater, stormwater) and those congeners that are significant contributors to Aroclor 1254, the primary aroclor present at the site. The list presented in the work plan was modified to incorporate coeluting congeners and a total of ~~45~~46 congeners will be measured for each station. The final list of congeners for passive sampler analysis, as well as the basis for their inclusion, is presented in Table ~~65~~65. Some of the congeners are currently noted as “TBA-to be added” to the current TTU method while others that exhibit coelution with other congeners are referenced as providing “SQ- Semi quantitative” analysis. Because of this coelution, it may not be possible to separate the congeners quantitatively, thus a semi-quantitative concentration will be reported.

A full scan of 130 congeners will be analyzed for a subset of 10 stations across the sample area. The 130 congeners represents the full list of congeners for which individual quantification has been developed. TTU analyzes a subset of the full 209 PCB congener list where they can positively separate the congeners to achieve an accurate quantification. To-date, TTU has not identified a need for attempting to quantify all 209 in environmental media. Two stations will be selected from each sampling subarea to evaluate the relative contribution of the ~~45~~46 selected congeners to the totals. The stations identified for full congeners list are identified in Section 2.

Table 65. Subset of PCB Congeners for Analysis

PCB Congener	Common in Bradford Island Media	Component of Aroclor	Congener of Ecological Concern	Addition since work plan	In Method	To be added (TBA) or semi-quant. (SQ)
6	•				•	
8	•		•		•	
11	•				•	
16	•				•	
18	•		•		•	
19	•				•	
44		•	•		•	
47			•		•	
49				•		TBA
52	•	•	•		•	
61	•			•	•	
65			•		•	
70	•	•			•	
74	•				•	
76	•				•	
83	•				•	
86	•			•		TBA
87	•	•			•	
90	•				•	
93	•				•	
95	•	•			•	
97	•	•				SQ
98	•			•		TBA
99	•	•			•	
100	•			•		TBA
101	•	•	•		•	
102	•			•		TBA
105	•	•	•		•	
108	•			•		SQ
110	•	•			•	
113	•				•	
115	•				•	
118	•	•	•		•	
119	•				•	
125	•			•		SQ
129	•					SQ
138	•	•	•		•	
147	•			•	•	
149	•	•			•	
153	•	•	•		•	
160	•					SQ
163	•	•			•	
168	•					SQ
180	•		•		•	
187	•		•		•	
193	•			•	•	
				TOTALs	35	11
						46

1.2.3. Statistical and Geospatial Analysis for Determination of Primary Source Material

As discussed in the Final Work Plan, a concentration threshold serves to identify those individual point locations that may indicate the presence of a primary source of PCBs.

In order to identify any individual points that may be indicative of a primary source, several statistical evaluations are being considered. One method being considered is to first evaluate the summed total subset of PCB congeners for each sample locations will and be statistically evaluate the data using ProUCL Version 5.1 software. A 90% UCL will could be calculated from the site wide data, and a multiplier of 10 will could be applied to the 90%UCL to establish a point threshold. The basis for a multiplier of 10 is predicated on previous studies where sediments adjacent to areas with NAPL were found to have porewater concentrations $\geq 1,000$ ng/L total PCBs. PCBs found in sediment porewater that were not associated with NAPL ranged in concentrations of 100 to 900 ng/L (Upal Ghosh, *personal communication*). This suggests that the presence of source material should result in concentrations at least an order of magnitude greater than non-source material for PCBs. Further, while Oregon DEQ guidance for establishing hot spot criteria is based on risk thresholds and point based evaluations, the application of a 10-times multiplier is used in hot spot threshold development, suggesting that concentrations of source material are an order of magnitude greater than materials that are not considered hot spots. Any individual points exceeding the established threshold will could be considered as indicative of a primary source of PCBs.

In additional to evaluating the site on a sum total basis, the congener results will be evaluated in ProUCL for potential mixture populations. This may be warranted if distinctly different aroclor populations represented by a smaller subset of congeners dominate over others. Q-Q plots will be utilized to visually assess the data for the presence of mixture populations along with other statistical tools as warranted. If population partitioning methods become necessary, those distinct populations may be summed separately prior to calculating site wide 90% UCLs for those separate populations and applying a 10-times multiplier.

Analysis for outliers in the dataset will also be performed using ProUCL software to elucidate whether any other samples could be considered indicative of a primary source.

Another statistical method being considered for establishing threshold concentrations indicative of source material is to apply a multiplying factor (e.g. 2x, 3x, 4x) to the standard deviation of the site average concentration. This multiplication of the standard deviation would then be applied to the calculated site average. Similar to establishing a site wide 90%UCL and using a 10x multiplying factor, this evaluation would also be done on a point basis for each sample locations. Evaluating either by the summed total subset of PCB congeners or groups would be considered. Individual points that are greater than the site average plus a multiplying factor of the standard deviation could be indicative of points containing PCB source material.

Data will also be looked at holistically through geospatial analysis with Kriging the data. While the statistical analysis aims to identify potential PCB sources on an individual point basis, Kriging will help

to provide analysis of PCB sources on a larger scale area basis. Kriging will be performed with ArcGIS, or a similar software package. Kriging will be used to relate individual points to potential larger spatial trends in the data. The results of Kriging will be evaluated cooperatively with individual threshold exceedances to determine potential areas indicative of a primary source. Specific geospatial methods will continue to be developed prior to receipt of data and coordinated with the Technical Advisory Group.

All of the above proposed statistical and geospatial methods will continue to be coordinated with the Technical Advisory Group prior to receipt of the data and throughout the data evaluation process.

The methods used for statistical and geotechnical evaluation along with the Results of this evaluation will be presented in the data evaluation report.

1.2.4. Temperature Monitoring to Assess Groundwater Influence

To help support interpretation of the passive sampling results, USACE will deploy temperature data loggers in conjunction with each individual passive sampler. Temperature will be recorded for the entire duration of deployment for each passive sampler. Identification of areas of groundwater discharge would allow the team to determine if groundwater discharging to the River (if it is) has an influence on results seen in the collocated passive samplers. Groundwater temperatures at the site are consistent (~9 - 13°C), but surface water (SW) varies from ~2 - 23°C. Differences in groundwater and surface water temperature at the groundwater /surface water interface may indicate discharge of groundwater to surface water.

1.2.5. Measurement Performance Criteria

Performance criteria specify the acceptable levels of uncertainty in measured data that can be used to support project decisions and achieve DQOs. Performance criteria for the analytical methods are specified in the laboratory procedures and are compliant with DoD QSM 5.1 unless otherwise noted. Any data which fall outside of these criteria must be justified, and the effects on decisions must be assessed.

1.3. Secondary Data Evaluation

No secondary data will be collected.

1.4. Project Overview and Schedule

Through project planning, the project team has agreed on the purpose of the project, the environmental questions that are being asked, and the environmental decisions that must be made. Project quality objectives have been developed specifying the type, quantity, and quality of data needed to ensure that project data can be used for the intended purpose to answer specific environmental questions, support environmental decisions, and determine technical activities that will be conducted. Table 6 provides a summary of the project tasks to be completed and ~~Table 7~~ ~~Table 8~~ describes the project schedule.

Table 76. Project Tasks

Plan, Prepare QAPP
<ul style="list-style-type: none">• Prepare and finalize QAPP.• Test deployment method/apparatus

Sampling Tasks
<ul style="list-style-type: none"> Deploy passive samplers and collocated temperature loggers Retrieve passive samplers and collocated temperature loggers
Analytical Tasks
<ul style="list-style-type: none"> Analyze LDPE for subset of congeners by GC-TQMS (Agilent 7890B) using SIM/SIM mode (EPA Method 1668c)
Quality Control Tasks
<ul style="list-style-type: none"> Analytical methods QC will comply with DoD QSM or laboratory SOPs as applicable.
Secondary Data
<ul style="list-style-type: none"> No secondary data will be collected.
Data Management Tasks
<ul style="list-style-type: none"> USACE Seattle Project Chemist will review and store analytical data.
Documentation and Records
<ul style="list-style-type: none"> Field notes will be recorded in a field notebook or on field log sampling sheets, then scanned and electronically stored. Field notes will contain the following: date and time of sample collection, weather conditions, sample identification number, type of sample, any procedural steps taken that deviate from those outlined in this QAPP. Laboratory analytical results will be stored.
Data Packages
<ul style="list-style-type: none"> 100% of data packages will be validated through Stage 2A or similar by the USACE Seattle Project Chemist.
Data Review Tasks
<ul style="list-style-type: none"> The laboratory performing analyses of samples will verify that all data are complete for samples received. Data will be validated using the principles of the <i>USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (2008)</i>. Validated data will be reviewed. Data usability will be assessed. Measurement performance criteria set in WP-QAPP checked. Data limitations will be determined. Data compared to Project Objectives.

Table 87. Estimated Project Schedule

Task #:Description	Start	Finish
Task #1: Plan, Prepare QAPP, Field test		
Draft and Final QAPP	October 2019	December 2019
Field test deployment method/apparatus	November 2019	December 2019
Task #2: Field Work		
Sample deployment	January 2019	2 weeks after start date
Sample retrieval	February 2019 (Minimum 28 days after deployment)	2 weeks after start date
Task #3: Review Lab Data and Prepare Report		
Laboratory analysis	Initiate upon receipt of samplers	1 month after receipt of samplers
Receive/Review Data Report and store electronically	Upon completion of laboratory analysis	Upon completion of laboratory analysis
Draft Data Evaluation Report	Upon completion of laboratory analysis	60 days after completion of laboratory analysis
Final Data Evaluation Report	Upon receipt of TAG comments	30 days after receipt of TAG comments

2. DATA GENERATION AND ACQUISITION

2.1. Sampling Tasks

2.1.1. LDPE Sampling Apparatus

The sampling material for the passive samplers will be LDPE sheets (10 cm x 10 cm x 25 μ m sheets) (or PE). This matrix is an established passive sampler material used for the measurement of PCBs in a variety of aquatic environments. The methods for both PE sheets and SPME samplers has been standardized (U.S. EPA, 2017) and a recent ESTCP project is currently working to refine the technology to improve the comparability of both field and laboratory methods across the industry. For the purposes of measuring PCB concentrations at the sediment-water interface, the PE samplers offer the advantages (over SPME samplers) of providing a higher surface to volume ratio at the sediment-water interface; they are durable for deployments in flowing water, and deployable in a variety of shapes and sizes. This allows the sampler apparatus to adapt to challenging environments while maximizing the amount of sampler exposed to the potential source areas.

Texas Tech University (TTU) will provide the PE sheets. Each sampler will be prepared with performance reference compounds (PRCs). PRCs are isotope labeled PCB analogues that are preloaded onto the PEs. The desorption rate constant of the PRCs will be used to approximate the absorption rate constant of the target analytes in order to quantify the equilibrium concentration.

A flexible, mesh envelope will secure the LDPE sheets. The mesh envelopes attach to the bottom of weighted pouches. The weighted pouch design ensures good contact between the PE sampler and the sediment surface and near bottom surface water, provides an anchor for the buoy lines, and allows for some interaction of the near-bottom water with the PE samplers. Weighted pouch construction includes wire mesh with openings of a minimum of 1/2" to allow water movement. The weight will be enclosed in the wire mesh pouch and will consist of 10 pounds of ~~1 be either 1.5 and 2"~~ steel ball bearings, ~~or large aggregate under a 6" x 6" x 2" steel weight.~~ The weighted pouch has a buoy line attached.

2.1.2. Temperature Data Loggers

Temperature will be recorded hourly at a minimum during the approximate 30 day deployment using Hobo pendant temp/alarm 64K and associated Hoboware Pro V.3.X software. Temperature sensors will be individually identified with a location specific site ID. Deployment and retrieval times for each individual data logger will be recorded in field notes. Temperature sensors will be secured inside the weighted bag immediately adjacent to the polyethylene sheets. Upon retrieval, temperature data will be transferred from the sensors and ~~average hourly temperatures will be calculated plotted for each~~ individual sample location.

2.1.3. Sampler Deployment and Retrieval

Sampling Stations

In order to support the DQOs listed in Section 1.2.1, passive samplers will be deployed at a total of ~~16370~~ locations along the northern and eastern shoreline of Bradford Island ~~(along with 5 passive samplers~~

deployed mid water column). The sampling area was based physical and chemical characteristics described in the work plan and included the following:

- Known location of debris
- Location of outfalls
- Downstream of debris or outfalls
- Areas with elevated PCB concentrations in sediment or clams
- Bathymetry

Station locations within the sampling area were selected using Visual Sampling Plan® (version 7.0 PNNL 2014) software based on a systematic random sampling design with a triangular grid and random start. Stations were placed at a density such that the maximum distance between sampling points was 7 to 14 m. Additional modifications to the sampling design following finalization of the work plan include the removal of stations that were randomly located in ~~unsamplable locations~~ unable to be sampled (e.g. upland points projecting in the sampling area) and the addition of sampling locations at the outer boundary of the sampling area to address TAG concerns to sample selected areas further offshore. Locations for the additional stations were based on the triangular grid established by VSP.

A table of all station locations with GPS coordinates for each location are presented in Appendix B. Figures of the sampling areas and selected stations is presented in Appendix C.

Deployment

Given the large number of samplers that need to be placed, along with the time and safety considerations associated with using a dive team for placement/retrieval, USACE will deploy and retrieve samples by boat and/or land (without divers).

Given the large number of samplers and duration required for deployment, LDPE samplers will be sent in batches to the field from TTU in order to minimize the time between sampler removal from the PRC loading solution and field deployment.

For boat deployment, the sampling vessel will maneuver to the pre-determined station coordinates using GPS. Because of the complexity of the river bed and the density of the stations, the sampling vessel will maneuver to each station using dynamic positioning. With dynamic positioning, the bow of the vessel would point into the current and work laterally across the sampling area. Alternatively, depending on field conditions, a three point anchor system may be used, with the vessel moving laterally across the sampling area using port and starboard anchors and moving upcurrent using the bow anchor. Once on station, the anchor lines will be tied off creating a stable sampling platform.

Once on station, each sampler – consisting of a passive sampler marked with the station number, temperature logger, and marker buoy marked with the station number, will be lowered to the bottom by hand using a deployment line. The deployment line will be an independent line that is weighted to decrease current influence and includes a triggered hook to allow release of the sampler and buoy line (See Appendix E for sampler schematics). Because the sampler needs to be placed in direct contact with the river bed and the river bed is uneven, the deployment line will be fitted with a real-time down-looking video camera to verify correct sampler placement and orientation on the sediment surface. Once the substrate has been considered suitable for deployment, the sampler will be released and station number, the time and date of sampler placement, coordinates recorded on a data sheet.

If the pre-determined station location is not suitable for sampler placement (e.g. based on visual observations of a boulder or large rocks), the sampler will be relocated to the nearest suitable location by moving laterally along the stern of the boat and alongside of the port and starboard side of the boat (a search area of approximately 10 ft². If a suitable station cannot be located within the search area, the station will be abandoned and a contingency location will be selected from the prepared list of preselected alternatives. Both primary and secondary contingency locations have been identified (see appendices B and C). Primary contingency points will be selected first from the set of alternate locations.

~~Deployment from land with a line to guide placement and assist in retrieval may occur for some samplers, particularly on the eastern tip. Given these deployment methods, accuracy will need to be managed and accounted for during data interpretation. The level of accuracy with determining the density of samplers given the field conditions is considered. Some sampling locations closest to the shoreline, particularly along the eastern tip of Bradford Island, may be difficult to access by boat for sampler deployment. As a contingency, deployment of samplers from land may be used for these shoreline locations. A handheld GPS will be used to determine land based deployment locations. However, given that deployment of these samplers will be offset by performing a land based deployment, accuracy relative to the target coordinates will be compromised relative to boat based deployment.~~

Retrieval

Samplers will remain in place for approximately 28 days to allow equilibration. Following the field exposures, samplers will be retrieved by hand from the sampling vessel. Marker buoys will be located

through visual observation. Once located, the samplers will be retrieved using the buoy lines. At the surface, the mesh envelopes will be separated from the weights and immediately processed for shipping to TTU. The date, time, and GPS locations will be recorded at the time of retrieval. Missing sampler or major discrepancies between the deployment and retrieval coordinates will be noted. Discrepancies between deployment and retrieval coordinates may indicate that the samplers were moved by currents during the 28 day deployment period. This uncertainty in location will be noted in the final results and accounted for in the data interpretation.

2.1.4. LPDE Sampler Field Processing

Upon recovery from the field, the PE, while still in the deployment device (e.g., stainless steel mesh), will be carefully cleaned (e.g., remove adhering sediment). The PE will be removed from the mesh and cleaned again with DI water, split into two sections as replicates and each replicate placed in pre-cleaned, amber, glass vials with a few drops of water for shipping. -All field processing of LDPE sheets will occur on a clean surface covered in aluminum foil inside the boat cabin. All samplers will be handled while wearing clean nitrile gloves. Subsequent lab processing will be conducted immediately (within 24 hours) of being received in the analytical laboratory.

Table 98. Methods, Sample Containers, Quantities, Volumes, Preservation, and Holding Times for Catch Basin Solids Samples

Analytes	Analytical Method	Container type/quantity	Preservation (all 4°C ± 2°C)	Minimum Mass per Sample	Holding Time	Number of field samples	Number of MS/MSD and Field Blanks	Total number of samplers containers ¹
Subset of PCB Congeners	GC-TQMS (Agilent 7890B) using SIM/SIM mode (EPA Method 1668c)	LDPE sheet (2 x 5*10 cm sheets)	A few drops of water	100 mg	Not extracted: <u>5 days at 4°C</u> Extracted: <u>1 year at -20°C</u>	170 (<u>163 –sediment;</u> <u>5 water column;</u> <u>2 backup</u>)	5	175

¹ The term “MS/MSD sample” encompasses two samples; therefore, an MS/MSD for PCBs, for example, would require four containers (two bottles for the MS and two bottles for the MSD).

2.1.5. Decontamination Procedures

New powder-free nitrile gloves will be donned at all times when handling ~~LPDE-LDPE~~ sheets. Upon retrieval of samplers, if any sediment is brought up with the sampling equipment, equipment will be rinsed from the side of the boat before bringing sampling device onto the boat deck.

2.1.6. Field Equipment Calibration, Maintenance, Testing and Inspection Procedures

No field equipment requires calibration, maintenance, testing and inspection. If any sampling procedures are changed to include use of field equipment, that information will be included in the field notes.

2.1.7. Supply Inspection and Acceptance Procedures

Inspection and acceptance of supplies and consumables will be conducted prior to field work in order to ensure that the appropriate type and quantity of supplies are brought to the field. Any supplies and consumables used in the sample collection process or instrument calibration will be inspected.

2.1.8. Field Documentation Procedures

Field documentation provides a permanent record of field activities and can be used, if necessary, to trace possible introduction of field sampling error.

Field notes will be maintained either in a bound logbook, or on field sampling log sheets. After fieldwork is complete, electronic copies will be made of the field notes and the electronic copies will be stored in the project files. All information pertinent to the sampling effort will be recorded in the field notes. Documentation in the field notes will be at a level of detail sufficient to explain and reconstruct field activities without relying on recollection by the field team members. The Field Sampling Lead has overall responsibility for accuracy and completeness of field notes. Each page/form will be consecutively numbered. All entries will be made in indelible ink and corrections will consist of lined-out deletions. As a minimum, the applicable items for the entry into the field notes are listed below.

General Information

- Date
- Start and finish times of work
- Weather conditions, including storm definition parameters:
 - ~~Time period and rainfall during preceding dry period~~
 - ~~Predicted storm duration and accumulation~~
 - ~~Afterwards, actual storm duration and accumulation~~
- Name and signature of person making entry
- Names of personnel present

Sampling Information

- Date and time of sample
- Location of sample

- Type of sample
- General river flow direction and velocity
- Water depth
- Sample identification number
- Associated QC samples
- Any unusual observations

2.1.9. Sample Delivery

Sample delivery procedures include packaging, labeling, and shipment to the laboratory. These procedures are designed (1) to preserve sample quality so that analyses will yield results representative of site conditions, (2) to protect and inform sample handlers, including shippers and laboratory personnel, and (3) to provide a paper trail to allow cross referencing of sample collection locations with analytical results.

All samples will be labeled with its own sample identification number and all other applicable information. Samples will be shipped to TTU at:

c/o Alex Smith, Brad Thornhill
 Department of Civil, Environmental and Construction Engineering
 Texas Tech University
 911 Boston
 Lubbock Texas 79409
 806.742.3523

2.1.10. Sample Custody

A sample is in “custody” if it is in the actual physical possession of authorized personnel or in a secure area that is restricted to authorized personnel. Custody procedures ensure data authenticity and defensibility. Chain of custody (CoC) forms will accompany sample containers during transit to the laboratory and be checked by the laboratory upon receipt.

2.1.11. Disposal of Investigative Derived Wastes

Personal protective equipment (PPE) for the sampling (consisting of Nitrile gloves) and other disposables used during sample preparation will be packaged in plastic garbage bags and disposed in a solid waste bin. All samples and chemical preservatives will be disposed of as per Texas Tech University hazardous material handling requirements.

2.2. Analytical Tasks

Once samples have been collected, they will be analyzed by the laboratories. The Project Chemist will validate the analytical data.

The following sections address all components of project-specific analytical measurements; method and laboratory-specific QC measurements; acceptance criteria; corrective actions; calibration procedures; equipment and supply maintenance; testing; and inspection requirements. Modifications to approved

procedures, alternate procedures, or additional procedures are to be pre-approved in writing by the Project Chemist.

2.2.1. Analytical Methods

See Table 9 for analytical methods that will be used for analysis of LDPE samples. All values detected between the LOD and the LOQ will be “J” flagged and reported as detections.

Table 9. Sample Locations, Media, Methods, Analytes of Interest, and Detection and Reporting Limits

<u>Sample Locations and Media</u>	<u>Method</u>	<u>Analytes</u>	<u>LOD^a</u>	<u>LOQ/RL^a</u>
River OU, LDPE	GC-TOMS (Agilent 7890B) using SIM/SIM mode (EPA Method 1668c)	Subset of total (46) PCB Congeners AND Total (130) PCB Congeners	<u>≤1 ng / g PE per congener</u>	<u>≤3.5 ng / g PE per congener</u>

^aDetection limits (LOD) and reporting limits (RL; also known as limit of quantitation (LOQ) are estimated and may change due to specific laboratory conditions, for example, dilutions.

The following isotopically labeled PCBs will serve as PRCs. The PRCs will be pre-loaded to the LDPE samplers to allow for equilibration correction during post processing analysis. See appendix A for laboratory SOPs related to PRC loading and analysis.

13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182

2.2.2. Analytical Instrument Calibration Procedures

Calibration procedures and instrumentation shall be consistent with the requirements of the methods.

2.2.3. Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures

Maintenance, testing, and inspection procedures shall be consistent with the requirements of the methods.

2.3. Quality Control Samples

Quality control (QC) samples are collected and analyzed for the purpose of assessing the quality of the sampling and analysis performed by the field personnel and the primary laboratory. The Project Chemist will coordinate selection of QC samples prior to each sampling event.

2.3.1. Field Quality Control Samples

2.3.1.1. Field Quality Control Samples

Field samples analyzed for the purpose of assessing the quality of sampling and analysis are to be submitted blind to the analytical laboratory and referred to as field QC samples.

2.3.1.2. Field Duplicates

No field duplicates will be taken for this sampling due to the small number of samples collected and limited budget.

2.3.1.3. Trip Blanks

No trip blanks will be taken for this sampling event as they are not necessary for the selected methods.

2.3.1.4. Field Blanks

Three field blanks will be taken and stored in the field and processed at time of retrieval in the same manner as field samples

2.3.2. Analytical Method Quality Control Samples

Method QC includes the analyses and activities required to ensure that the analytical system is in control prior to and during an analytical run. Method QC requirements for this project include the following: method blanks, surrogate spikes, matrix spikes/matrix spike duplicate pairs, and laboratory control samples.

2.3.2.1. Method Blanks

Method blanks are composed of organic/analyte-free water processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. Method blanks verify that the measurement system is free of contamination.

2.3.2.2. Laboratory Control Samples (LCS)

Laboratory control sample (LCSs) are composed of organic/analyte-free water spiked with verified amounts of analytes. They are generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system. The LCS is analyzed in the same manner as a sample, including preservation.

2.3.2.3. Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSD samples are used to evaluate matrix interference and to determine laboratory accuracy and precision. ~~Five MS/MSD samples have been identified by TTU for this effort. For methods that require MS/MSD, One MS/MSD per method will be taken for this sampling event, at a location that is up to the discretion of the field sampling team.~~

2.3.2.4. Surrogates

Surrogates are substances with properties that mimic the analyte of interest. A surrogate is unlikely to be found in environment samples, and is therefore added to them for quality control purposes.

3. ASSESSMENT AND OVERSIGHT

Laboratory and field operations have established policies and procedures, and they designate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the quality system or technical operations have been identified. Both field and laboratory operations shall follow all corrective action requirements in methods and SOPs.

The following laboratory documentation is to be made accessible to the USACE Project Chemist. Corrective actions may be required, at the request of USACE, for the following conditions:

- Laboratory Procedures
- QC data outside the defined acceptance windows for precision or accuracy
- Blanks or Laboratory Control Samples (LCS) that contain contaminants above acceptable levels stated in the Data Quality Objectives
- Undesirable trends in spike or surrogate recoveries or RPD between spiked duplicates
- Unusual changes in method detection limits
- Deficiencies identified during internal or external audits or from the results of performance

The following corrective actions should be taken for common problems:

Incoming Samples - Problems noted during sample receipt are to be documented. The USACE Project Chemist is to be notified for problem resolution.

Sample Holding Times - If a maximum holding time is or may be exceeded by the laboratory, the USACE Project Chemist must be notified for problem resolution. The USACE Project Chemist may require re-sampling for the requested parameters.

Instrument Calibration - Sample analysis may not proceed until initial calibrations meet method criteria. Calibrations must meet method time requirements or recalibration must be performed. Continuing calibrations that do not meet accuracy criteria should result in a review of the calibration, rerun of the appropriate calibration standards, and reanalysis of samples affected back to the previous acceptable calibration check.

Limit of Quantitation (LOQ) - Appropriate sample clean-up procedures must be employed to attempt to achieve the practical quantitation limits as stated in the method. If difficulties arise in achieving these limits due to a particular sample matrix, the laboratory should notify the USACE Project Chemist of the problem for resolution. Dilutions are to be documented in the case narrative along with the revised practical quantitation limits for those analytes directly affected. Analytes detected above the method detection limits (MDLs) but below the practical limit(s) of quantitation are to be reported as estimated values and qualified "J".

Method Quality Control - Results related to method QC, including blank contamination, duplicate measurement reproducibility, MS/MSD recoveries, surrogate recoveries, LCS recoveries, and other method-specified QC measures are to meet the laboratory's SOPs and PQOs specified in this plan. Otherwise, the affected samples may be reanalyzed and/or re-extracted and reanalyzed within method-required holding times to verify the presence or absence of matrix effects. In order to confirm matrix effects, QC results must observe the same direction and magnitude (ten times) bias. The USACE Project Chemist should be notified as soon as possible to discuss appropriate corrective action.

Calculation Errors - Reports must be reissued if calculation and/or reporting errors are noted with any given data package. The case narrative is to state the reason(s) for re-issuance of a report.

4. DATA MANAGEMENT AND DOCUMENTATION

4.1. QAPP

An electronic copy of the QAPP (including appendices) will be stored in USACE project files and provided to the Technical Advisory Group.

4.2. Final Report

Upon completion of the sampling event and receipt/review of the validated data, USACE will prepare a final report. The report may be issued separately, or as an appendix to a future report that addresses source control. The report will include the following:

- Narrative and timeline of project activities
- Summary of sampling, chemical testing, and any deviations from the QAPP
- Analytical data summary and discussion
- Figures, tables, and appendices

The appendices will include field logs, laboratory analytical reports, data validation reports, and data summary tables with associated validation flags.

4.3. Laboratory Documentation (Data Package Deliverables)

4.3.1. Data Package Deliverables

The analytical data packages from the laboratories will be provided to the USACE Seattle Project Chemist as Stage 4 or similar deliverables. The analytical data packages will be validated to Stage 2A or similar by the Project Chemist for 100% of all samples analyzed by the laboratory.

4.3.2. Electronic Data Reporting Formats

~~Due to the small sampling scope of this project, laboratory data will be accepted as a report in PDF format. Additional electronic data deliverables (EDD) are not required.~~ The laboratory data will be provided in Microsoft Excel format. A copy of the laboratory data will be provided to the Technical Advisory Group upon completion of the data validation.

5. DATA REVIEW, VERIFICATION, AND VALIDATION

Data review is the process by which data are examined and evaluated to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. It includes verification, validation, and usability assessment. This process ensures the review activities produce scientifically sound data that are of known and documented quality and meet PQOs used in making environmental decisions.

5.1. Review of Laboratory Data

All laboratory data packages will include raw data necessary for full validation. Analytical data packages will be validated to Stage 2A or similar by the USACE Seattle Project Chemist for 100% of all samples analyzed by the contracted laboratory (TTU).

Three distinct evaluative steps will be used to ensure that project-specific data quality needs are met:

- Data Verification (review for completeness) – Confirmation by examination and provision of objective evidence that the specified requirements (sampling and analytical) have been completed.
- Data Validation – Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Validation is a sampling and analytical process that includes evaluating compliance with method, procedure, or contract requirements and extends to evaluating against criteria based on the quality objectives developed in the QAPP (e.g., the QAPP measurement performance criteria). The purpose of validation is to assess the performance of the sampling and analysis processes to determine the quality of specified data. Data Validation Reports will be generated for each sampling event.
- Data Usability Assessment – Determination of the adequacy of data, based on the results of validation and verification, and professional judgment by the Project Chemist, for the decisions being made. The usability step involves assessing whether the process execution and resulting data meet project quality objectives documented in the QAPP.

Data review will be based on laboratory-specific SOPs conforming to the method and applying the principles of the EPA National Functional Guidelines for Organic and Inorganic Data Review (EPA 2008). If significant deviations arise as a result of initial verification and validation, the level of review will be elevated in order to determine the source and impact of deviations.

5.2. Data Verification and Validation Stages

Data validation and verification stages described below are in accordance with US EPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA QA-R-08-005; 2009).

5.2.1. Stage 1

Verification and validation begins with Stage 1 checks of the laboratory analytical data package consisting of compliance of sample receipt conditions, sample characteristics (e.g., percent moisture), and

analytical results (with associated information). The following minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 1 validation label:

- (1) Documentation identifies the laboratory receiving and conducting analyses, and includes documentation for all samples submitted by the project or requested for analyses.
- (2) Requested analytical methods were performed and the analysis dates are present.
- (3) Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result (and the uncertainty of each result and clear indication of the type of uncertainty reported if required).
- (4) Requested target analyte result units are reported.
- (5) Requested reporting limits for all samples are present and results at and below the project-specific reporting limits are clearly identified (including sample detection limits if required).
- (6) Sampling dates (including times if needed), date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH and temperature) are documented.
- (7) Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture) to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.2. Stage 2A

Stage 2A validation builds on the validation conducted in Stage 1. Stage 2A validation of the laboratory analytical data package consists of the Stage 1 validation plus the verification and validation checks for the compliance of sample-related QC. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 2A Validation label:

- (8) Requested methods (handling, preparation, cleanup, and analytical) are performed.
- (9) Method dates (including dates, times and duration of analysis for radiation counting measurements and other methods, if needed) for handling (e.g., Toxicity Characteristic Leaching Procedure), preparation, cleanup and analysis are present, as appropriate.
- (10) Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compounds (DMC) recoveries, laboratory control sample (LCS) recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries) are provided and linked to the reported field samples (including the field quality control samples such as trip and equipment blanks).
- (11) Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes) have been added, as appropriate.

(12) Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.

(13) Frequency of QC samples is checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch).

(14) Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.3. Stage 2B

Stage 2B validation builds on the validation conducted in Stage 2A. Stage 2B validation of the laboratory analytical data package consists of the Stage 2A validation plus the verification and validation checks for the compliance of instrument-related QC. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 2B Validation label:

(15) Initial calibration data (e.g., initial calibration standards, initial calibration verification [ICV] standards, initial calibration blanks [ICBs]) are provided for all requested analytes and linked to field samples reported. For each initial calibration, the calibration type used is present along with the initial calibration equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the initial calibration curve are present, along with their associated percent recoveries, as appropriate (e.g., if required by the project, method, or contract). For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present.

(16) Appropriate number and concentration of initial calibration standards are present.

(17) Continuing calibration data (e.g., continuing calibration verification [CCV] standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present.

(18) Reported samples are bracketed by CCV standards and CCBs standards as appropriate.

(19) Method specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods).

(20) Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatography-mass spectroscopy [GC-MS] tunes have been run every 12 hours).

(21) Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.4. Stage 3

Stage 3 validation builds on the validation conducted in Stage 2B. Stage 3 validation of the laboratory analytical data package consists of the Stage 2B validation plus the recalculation of instrument and sample results from the laboratory instrument responses, and comparison of recalculated results to laboratory reported results. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 3 Validation label:

- (22) Instrument response data (e.g., GC peak areas) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, matrix spikes, matrix spike duplicates, LCS, and method blanks as well as calibration data and instrument QC checks (e.g., tunes).
- (23) Reported target analyte instrument responses are associated with appropriate internal standard analyte(s) for each (or selected) analyte(s) (for methods using internal standard for calibration).
- (24) Fit and appropriateness of the initial calibration curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the initial calibration curve for each (or selected) analyte(s) from the instrument response.
- (25) Comparison of instrument response to the minimum response requirements for each (or selected) analyte(s).
- (26) Recalculation of each (or selected) opening and closing CCV (and CCB) response from the peak data reported for each (or selected) analyte(s) from the instrument response, as appropriate.
- (27) Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated initial calibration response for each (or selected) analyte(s).
- (28) Recalculation of percent ratios for each (or selected) tune from the instrument response, as appropriate.
- (29) Compliance check of recalculated percent ratio for each (or selected) tune from the instrument response.
- (30) Recalculation of each (or selected) instrument performance check (e.g., instrument blanks,) from the instrument response.
- (31) Recalculation and compliance check of retention time windows (for chromatographic methods) for each (or selected) analyte(s) from the laboratory reported retention times.
- (32) Recalculation of reported results for each reported (or selected) target analyte(s) from the instrument response.
- (33) Recalculation of each (or selected) reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries) from the instrument response.

(34) Each (or selected) sample result(s) and spike recovery(ies) are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

Note: Selection of analytes, spikes, and performance evaluation checks for the Stage 3 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including (but not limited to) the type of verification and validation being performed (manual or electronic), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

5.2.5. Stage 4

Stage 4 validation builds on the validation conducted in Stage 3. Stage 4 validation of the laboratory analytical data package consists of the Stage 3 validation plus the evaluation of instrument outputs. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 4 Validation label:

(35) All required instrument outputs (e.g., chromatograms, mass spectra) for evaluating sample and instrument performance are present.

(36) Sample results are evaluated by checking each (or selected) instrument output (e.g., chromatograms, mass spectra) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences).

(37) Each (or selected) instrument's output(s) is evaluated for confirmation of non-detected or tentatively identified analytes.

Selection of instrument outputs for the Stage 4 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including, but not limited to, the type of verification and validation being performed (electronic or manual), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

5.3. Data Verification and Validation Stages

A data validation report will be generated by the USACE Chemist that encompasses the results of the manual review of private lab data. The data validation report will be an appendix to the Final Report. Professional judgment shall be used when deciding if qualification of data is applicable. When professional judgment is applied, the rationale shall be provided. Tables of qualified data and the reasons for qualification will also be included in the data validation report.

Qualifiers will be added to data during the review as necessary. Qualifiers applied to the data as a result of the review are as follows:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit. The data are usable for decision-making purposes.
- UJ Indicates the compound or analyte was analyzed for but not detected. Due to a quality control deficiency identified during data validation, the value reported may not accurately reflect the sample quantitation limit. The associated value is considered estimated, but the data are generally usable for decision-making purposes.
- J Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. False positives or false negatives are unlikely to have been reported and the data are generally usable for decision-making purposes.
- J+ Data are qualified as estimated with a high bias. False positives are likely to occur but the data are generally usable for decision-making purposes.
- J- Data are qualified as estimated with a low bias. False negatives are likely to occur but the data are generally usable for decision-making purposes.
- R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Note: It is possible that J-qualified data are not suitable for some purposes. For example, a J-qualified concentration with a low bias that is just below a screening value may not be usable to determine whether the analyte concentration is above or below the screening value. The effect of the use of qualified data on the decision-making process must be evaluated as part of the “reconciliation with user requirements” process.

5.4. Usability Assessment

The Project Chemist will evaluate overall precision, accuracy, completeness, representativeness, comparability, and sensitivity of the sampling data; including an assessment of the overall usability of the data and describing any limitations on its use. The Project Chemist will summarize any audit information, indicating corrective actions taken. This information will be part of the data validation report, which is an appendix to the Final Report.

6. REFERENCES

U.S. Army Corps of Engineers. 2019. *Final Work Plan for Passive Sampling at River OU, Bradford Island, Cascade Locks, Oregon*. September 30, 2019.

U.S. Environmental Protection Agency. 2009. *Intergovernmental Data Quality Task Force Uniform Federal Policy for Quality Assurance Project Plans Guidance*.

U.S. Environmental Protection Agency. 2008. *Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*.

URS. 2012. *Upland and River Operable Units Remedial Investigation Report, Bradford Island, Cascade Locks, Oregon*.